

ASH DEPOSITION BEHAVIOR OF A COAL, A CLEAN COAL, AND A CHAR IN A DROP TUBE FURNACE

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INTRODUCTION

This work was undertaken as part of a larger study to evaluate the combustion characteristics of chars derived from Illinois coals under mild gasification (MG) conditions. The principle product (60 to 70 percent by weight) of MG processes is a char that must be effectively utilized to improve the overall economics of the process. During the past several years, one of the major research activities at the Illinois State Geological Survey has been to examine the suitability of using MG char as a fuel, alone or as coal-char blend, in typical industrial pulverized-coal (PC) boilers. The physical and chemical characteristics and reactivity of laboratory- and pilot plant-prepared MG chars have been reported (1-4).

In this paper preliminary results of a study to evaluate the ash deposition behavior of an MG char under conditions representative of PC boilers are presented. The results are compared to those obtained with the raw coal and a physically cleaned coal. Ash deposition tests were performed in a laminar flow (drop tube) furnace.

EXPERIMENTAL

Sample Preparation - The clean coal and the char were made from an original sample of the Herrin coal seam (IL No. 6) that was provided by the Illinois Basin Coal Sample Program, identified as IBC-101 (5). The parent sample represented the product from the mines preparation plant. The analysis of the coal is given in table 1.

The clean coal was prepared from a 200x400 mesh size fraction of the original coal in a Denver model D-2 batch flotation system. Approximately 150 grams of coal was combined with four liters of water in the flotation cell. A flotation agent (2-ethyl hexanol) was added at an equivalence of 6 lbs/ton. Additional ash was removed by repeating the procedure using the clean coal as the starting material. The clean coal was vacuum filtered, dried in a vacuum oven at 80 °C, and stored under nitrogen to prevent oxidation. The analysis of the product sample is given in table 1. The amount of ash in the clean coal is 6.4% which corresponds to a 34% decrease compared to the parent (200x400 mesh) coal. However, the clean coal retains almost 87% of the sulfur present in the original coal. This is because 72% of the sulfur in the parent coal is in the form of organic sulfur which is not removed by flotation methods.

A 5.1 cm ID batch fluidized-bed reactor system was used for the production of the char. The sample used for char production was a 28x100 mesh size fraction of the coal. In each run, about 200 grams of the coal was fluidized by nitrogen flowing at 6 l/min and heated according to a multi-step heating procedure to minimize agglomeration of coal particles in the reactor. The final temperature and final soak time were adjusted to produce three chars with volatile matter contents of about 7, 12, and 15%. The 7% volatile char was selected for ash deposition studies because it had higher ash content (14.1%, see table 1) than the other chars. The final temperatures and soak times for the char were 600 °C and 60 mins. After the final soak, the samples were cooled under

nitrogen purge and stored under nitrogen.

Ash Characteristics - The ash compositions (major and minor oxides) were determined by routine methods, and the ash fusability temperatures of the coal, clean coal, and char were determined by the ASTM D1857 method. These data were used to calculate silica ratio, base/acid ratio, T_{cv} (temperature at critical viscosity), T_{250} (temperature at which the slag has a viscosity of 250 poise), the viscosity of slag at 2600 °F and the slagging index and fouling index.

Ash Deposition Studies - Ash deposition tests were conducted in a drop tube furnace (DTF) located at the University of North Dakota's Energy and Environmental Research Center (UNDEERC). The DTF is described elsewhere (6). A brief description of the ash deposition probe follows.

The water-cooled ash deposition probe, shown in figure 1, consists of a 2.2 cm OD stainless steel tube with a 3.8 cm OD diameter cap. A machined, boiler-steel substrate plate (3.8 cm diameter by 0.64 cm thick) prepared from 1040 carbon steel, supplied by the Babcock and Wilcox Company, is attached to the top of the probe. The substrate plate temperature can be maintained between 350 to 540 °C to simulate a boiler heat transfer surface, by adjusting cooling water and the tightness of the screws that hold the substrate to the probe. The temperature is monitored by a Type K thermocouple in contact with the plate. The substrate plates were polished with SiC polishing discs starting at 120 grit and proceeding to 600 grit. The substrates were oxidized at 400 °C for 20 hours in air to produce an oxide layer on the steel.

In each test, the drop tube furnace was stabilized at 1500 °C and the nominal gas flow rates of 1 l/min primary gas flow and 3 l/min secondary gas flow. The sample feeder was loaded with the desired sample, and the feed rate adjusted to give approximately 0.2 g/min. After the test run, the probe was allowed to cool and the substrate with the ash deposit removed and stored pending strength tests on the ash deposit. The strength of the deposits formed in the DTF were measured using a strength testing apparatus. The strength testing apparatus is described elsewhere (6).

RESULTS

Ash Characteristics - Analyses of the ash from these samples showed only small differences between the coals and char (table 2). The cleaning process removed soluble sulfate minerals, and about half the calcite. The clay minerals and quartz were retained in the clean coal. The temperature for which the ash achieves the desired viscosity of 250 poise (T_{250} , table 3) ranged from 2030 °F (clean coal) to 2115 °F (char). Experience indicates values below 2600 °F are desirable (7).

Slagging indices for the three samples fell in the medium range, according to Attig and Duzy (8), while the fouling index was high for the feed coal and char samples and medium for the clean coal (table 3).

Ash deposition studies - The results of the ash deposition tests are shown in table 4. The ash weight was normalized to a feed rate of 0.13 g/min. This gave a basis to compare the different tests. Clean coal produced about 50% more deposit than the parent coal (figure 2). However, the parent coal and clean coal gave deposit weight curves with similar slopes. Ash deposit weights of the char were substantially higher than either coal or clean coal.

The deposit weight for the clean coal increased faster than that of the parent coal in spite of the fact that it contains about two-thirds the ash of coal (9.7% vs. 6.4%). Without a detailed mineral analysis of the deposits, it is difficult to determine the cause of this behavior. Because the cleaning process removed a portion of the original

mineral matter (particularly calcite, see table 2), altering the composition of the remaining ash, the new composition may have resulted in a "stickier" ash with a higher melting point than the original ash. The ash deposition behavior of the char is consistent with its high ash content (14.1%).

The deposit growth rate curves were used to calculate sticking coefficients. The sticking coefficient (SC) is defined as:

$$SC = \text{rate of ash deposition/rate of firing of ASTM ash}$$

where "ASTM ash" is the ash yield of the fuel as determined by the standard ASTM proximate analysis procedure, or in this study, by TGA analysis of the fuel. The sticking coefficient is a normalized measure of how much ash in the fuel is sticking to the substrate plate of the ash deposition probe, and has a value between zero and one. A value of zero indicates that none of the ash is sticking; a value of one indicates that all of the ash is sticking. The higher the value of the sticking coefficient, the more slagging and fouling are likely to occur.

The deposit rates were calculated from best-fit curves that were determined using the deposit weight and time (table 4). For the parent coal and clean coal, a quadratic equation was fitted to the deposit weight data. It was not possible to obtain a meaningful quadratic equation to fit the data for the char. The instantaneous deposit rates were divided by the ASTM ash firing rate to give the sticking coefficients. The data indicate that the clean coal deposits grew at a faster rate than the parent coal (figure 3). While it was not possible to determine a numerical sticking coefficient for char, the normalized deposit weight data presented in table 4 show that the sticking coefficient for char is high, indicating that the deposits from the char grew much faster than those of the coal samples.

The differences observed in the sticking coefficients for the parent coal and clean coal are likely related to the association of the inorganic constituents in the coal. The inorganic components in the clean coal occur within and/or with carbonaceous particles because the cleaning process removes much of the extraneous (or carbon free) mineral grains. During combustion, the inorganic components associated with coal particles are subjected to higher temperatures than extraneous mineral grains. As a result of the higher temperatures, more of the particles melt and are sticky when they reach the deposition probe, producing a higher sticking coefficient. It is also possible that the included minerals are collected on the surface of the extraneous mineral particles (those deposited on the probe) and carry off the probe by the extraneous mineral particles.

The deposit strength curves are shown in figure 4. Clean coal has a gradual slope to the deposit strength curve and is lower in strength than the coal. The parent coal has an initial slope similar to that of clean coal, but the strength increases rapidly near the top of the deposit. The deposit strength of char exceeded the maximum capacity of the test apparatus (100 psi) and was not included in figure 4.

The difference in the strength of the deposits produced from the parent and cleaned coals is probably due to the types of ash particles in deposits. The strength of the clean coal deposits was lower than the other samples because some of the reactive, liquid producing ash components were removed by cleaning. For example, during the cleaning process a significant amount of pyrite is removed. The iron in pyrite, when combined with aluminosilicates in a combustion environment, produces low melting-temperature phases that are responsible for greater deposit strengths. The greater amount of iron present in the parent coal probably caused the parent coal's stronger deposits. The deposits produced from the clean coal most likely did not contain sufficient fluxing agents such as iron to develop high strength.

Physically, the deposits left by the clean coal were not molten whereas the deposits left by the coal were molten near the top. The deposits left by the char were molten not only at the top but also down the center of the deposit. Melting of deposits increases their strength; this explains the high strength of the char deposits.

CONCLUSIONS

The ash deposition behavior of an Illinois Herrin (No. 6) coal, and of a clean coal product, and a char derived from the parent coal was evaluated. The deposits were collected on a water-cooled stainless steel probe located inside an entrained flow reactor operating at 1500 °C and 20% oxygen. Standard ASTM analyses showed only small differences between the composition of the coal and char ashes; however, the clean coal had substantially lower soluble sulfate minerals and calcite than the coal. Ash deposition studies in a DTF showed that although the clean coal produced a larger deposit that grew at a faster rate than that of the original coal, the clean coal deposits were weaker than those of the coal and showed evidence of partial melting. The deposits from the char grew much faster than that of either the coal or clean coal. Due to extensive melting, the deposits formed by the char were much stronger than those of either the parent coal or clean coal.

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Table 1. Proximate and ultimate analysis for samples (dry basis)

Particle Size Mesh	270x400 Parent Coal	200x400 Clean Coal	270x400 Char
Moisture	2.9	1.6	0.9
<u>Proximate, wt%^a</u>			
Volatile Matter	41.8	41.5	7.3
Fixed Carbon	48.5	52.2	78.9
H-T Ash	9.7	6.3	13.8
<u>Ultimate, wt%^b</u>			
Hydrogen	5.2	5.5	2.2
Carbon	69.5	73.3	78.2
Nitrogen	1.5	1.8	2.2
Oxygen ^c	9.5	9.8	1.1
Sulfur	4.6	4.0	2.9
BTU/lb	12674	13293	12791
lb SO ₂ /MMBTU	7.2	6.0	4.6

^a TGA analysis, ^b LECO CHN 600 analyzer, ^c determined by difference

Table 2. Ash composition

Oxide	Parent Coal*	Clean Coal	Char
<u>Ash Analyses (% of ash)</u>			
SiO ₂	48.60	49.30	48.20
Al ₂ O ₃	17.49	17.81	17.36
Fe ₂ O ₃	18.22	19.53	18.08
CaO	4.89	2.20	4.76
MgO	0.99	1.31	0.98
K ₂ O	2.22	2.37	2.22
Na ₂ O	1.47	1.11	1.50
TiO ₂	0.89	1.34	0.90
P ₂ O ₅	0.27	0.14	0.26
MnO ₂	0.05	0.04	0.06
SrO	0.04	0.05	0.04
BaO	0.04	0.05	0.04
SO ₃	4.19	1.94	4.29
Silica ratio	68.85	68.15	66.93
Base/acid	0.41	0.39	0.41

*Mineral matter in IBC-101: 2.6% quartz, 0.5% calcite, 2.1% pyrite and Marcasite, 3.3% kaolinite, 2.4% illite, and 2.1% expandable clay.

Table 3. Ash fusability temperatures

	Parent Coal	Clean Coal	Char
<u>Ash Fusion ('F, reducing)</u>			
Initial def.	2055	2030	2115
Softening	2140	2140	2185
Hemispheric	2225	2245	2260
Fluid	2310	2350	2330
<u>Empirical ash properties</u>			
T ₂₅₀ , 'F	2425	2480	2460
T _{cv} , 'F	2584	2558	2584
Slag viscosity (poise) @2600 'F	105	125	106
Slagging index	1.75	1.53	1.20
type	med.	med.	med.
Fouling index	0.60	0.43	0.62
type	high	medium	high

Table 4. Ash deposition weights

Sample	Time (min)	Coal fed (g)	Ash (g)	Normalized ash (g)
Char	10	0.78	0.0619	0.1032
	20	2.70	0.2669	0.2570
	30	3.68	0.2618	0.2775
Parent coal	10	1.48	0.0488	0.0429
	20	2.24	0.0650	0.0754
	30	3.52	0.1029	0.1140
Clean coal	10	0.90	0.0463	0.0669
	20	3.81	0.1438	0.0981
	30	2.83	0.1070	0.1475

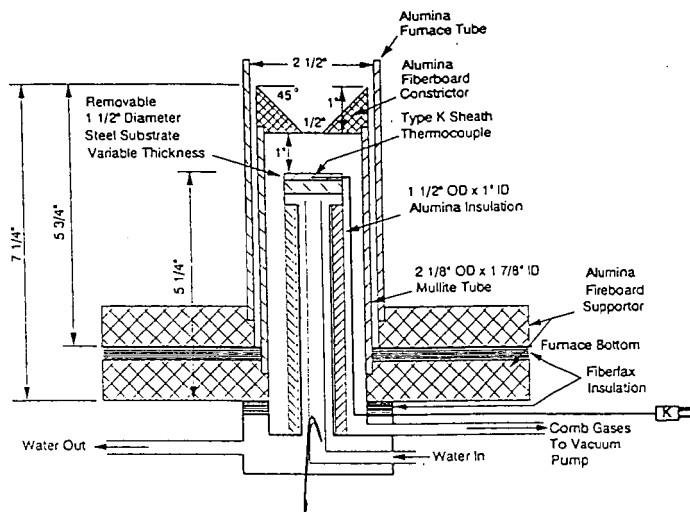


Figure 1. Ash deposition probe.

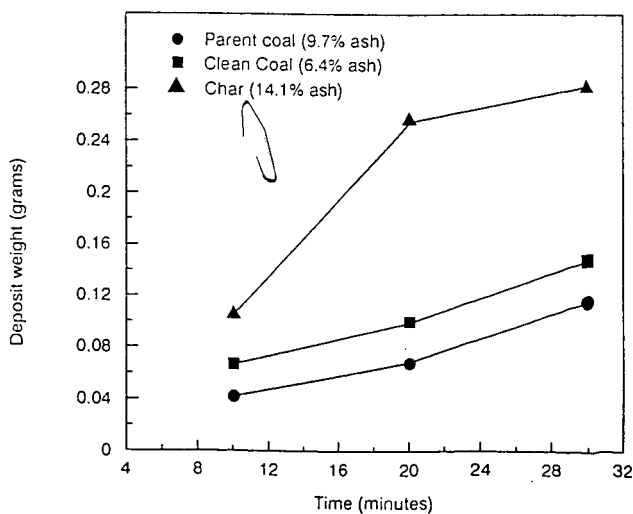


Figure 2. Deposit growth rates for fuels ($T = 1500^{\circ}\text{C}$)

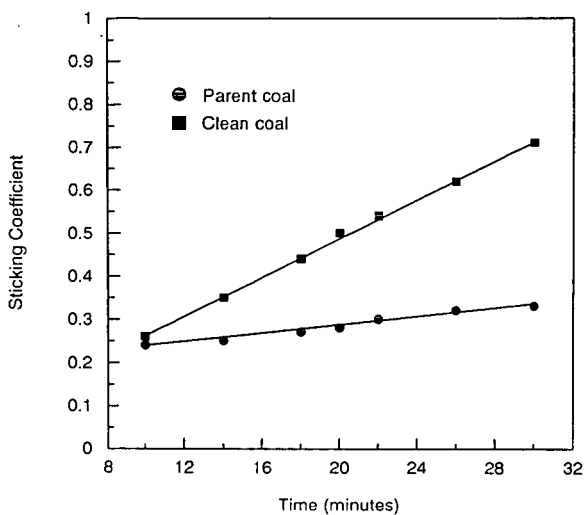


Figure 3. Sticking coefficients for coal samples.

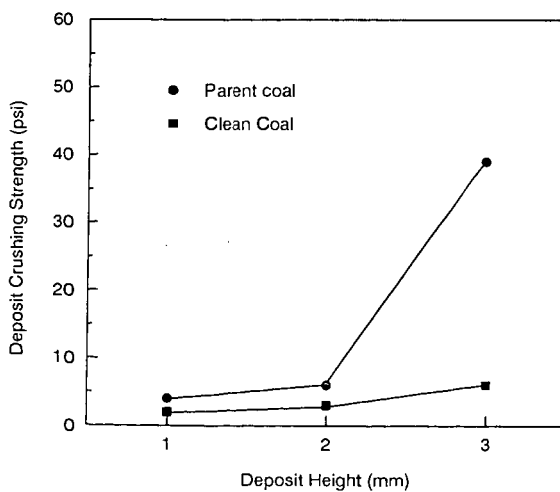


Figure 4. Deposit crushing strength for coal samples.